

FINAL YEAR PROJECT REPORT

ON

**Study of Dynamic Behavior of Ethyl Acetate  
Reactive Distillation Column Using ASPEN PLUS.**

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**CERTIFICATE**

This is to certify that the thesis entitled, “Study of Dynamic Behavior of Ethyl Acetate Reactive Distillation Column Using ASPEN PLUS” submitted by Susheel Kumar Javaji in partial fulfillments for the requirements for the award of Bachelor of Technology Degree in Chemical Engineering at National Institute of Technology, Rourkela (Deemed University) is an authentic work carried out by him under my supervision and guidance.

To the best of my knowledge, the matter embodied in the thesis has not been submitted to any other University / Institute for the award of any Degree or Diploma.

Date:

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## **ACKNOWLEDGEMENT:**

I would like to express my sincere gratitude to the Department of Chemical Engineering, N. I. T. Rourkela for helping me to complete my project successfully. I take great pleasure in presenting this report on my project “Study of Dynamic Behavior of Ethyl Acetate Reactive Distillation Column Using ASPEN PLUS.” and in expressing my deep regards towards those who offered invaluable guidance in hours of need. I would like to thank all other faculty members for their support and help whenever needed. They were always there to help me.

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**J Susheel Kumar**

**10500021**

## CONTENTS:

Abstract	i
List of figures	ii
<b>Topic</b>	<b>Page No.</b>
CHAPTER 1: INTRODUCTION	1
1.1 Properties and uses of ethyl acetate	2
1.2 Production of Ethyl acetate	3
1.3 Back ground of Reactive distillation process	4
1.4 Background of ASPEN PLUS software	5
1.5 Background of Aspen dynamics	6
CHAPTER 2: LITERATURE REVIEW	7
CHAPTER 3: MODELING	10
3.1 Component material balance	11
3.2 Energy balance	12
3.3 Equilibrium relationships	12
CHAPTER 4: STEADY STATE	13
4.1 Single feed input data	14
4.2 Double feed	14
4.3 Effect of reflux ratio on ETAC mol fraction	15
4.4 Effect of bottoms rate on ETAC mol fraction	15
CHAPTER 5: DYNAMICS	16
CHAPTER 6: RESULTS	18
6.1 Conclusion	27
CHAPTER 7: REFERENCES	28

## **ABSTRACT:**

Reactive distillation (RD), the combination of chemical reaction and distillation in a single unit operation, has proven to be advantageous over conventional process systems consisting of separate reactor and distillation units. But the dynamic behavior of process is difficult to study. In this thesis, a reactive distillation column for ethyl acetate production has been created in ASPEN user interface. Steady state simulations are done in ASPEN user interface and the effect of reflux ratio on the composition of ethyl acetate in the distillate is studied. In ASPEN DYANAMICS the composition control studies for ethyl acetate purity has been studied both in the distillate and in the bottoms.

## LIST OF FIGURES:

Fig num	Name	Page no
1	Single feed flow sheet in ASPEN	19
2	Single feed composition profile	19
3	Optimum tray for second feed	20
4	Double feed flow sheet in ASPEN	20
5	Double feed composition profile	21
6	Effect of Reflux ratio on the ETAC composition	21
7	Effect of Bottoms rate on the ETAC composition	22
8	Composition control for ethyl acetate in Distillate	22
9	Composition control for ethyl acetate in bottoms	23
10	Response of controller for XD when $K_c=0.5$	23
11	Response of controller for XD when $K_c=1$	24
12	Response of controller for XD when $K_c=1.5$	24
13	Response of controller for XD when $K_c=2$	25
14	Response of controller for XB when $K_c=1$	25
15	Response of controller for XB when $K_c=0.5$	26
16	Response after tuning the controller for XB	26

# CHAPTER-01

# INTRODUCTION

Ethyl acetate is the organic compound with the formula  $\text{CH}_3\text{COOC}_2\text{H}_5$ . This colorless liquid has a characteristic, pungent smell like certain glues or nail polish removers, in which it is used. Ethyl acetate is the ester from ethanol and acetic acid; it is manufactured on a large scale for use as a solvent. Ethyl acetate is a moderately polar solvent that has the advantages of being volatile, relatively non-toxic, and non-hygroscopic. It is a weak hydrogen bond acceptor, and is not a donor due to the lack of an acidic proton. Ethyl acetate can dissolve up to 3% water and has a solubility of 8% in water at room temperature. It is unstable in the presence of strong aqueous bases and acids. It is Soluble in most organic solvents, such as alcohol, acetone, ether and chloroform.

### 1.1 Properties and Uses:

Molecular formula	$\text{CH}_3\text{COOC}_2\text{H}_5$
Molar mass	88.105 g/mol
Density	0.897 g/cm <sup>3</sup>
Melting point	-83.6 °C
Boiling point	77 °C
Viscosity	0.426 cp at 25°C

Ethyl acetate has a wide range of applications, across many industries, including:

**Surface coating and thinners:** Ethyl acetate is one of the most popular solvents and finds wide use in the manufacture of nitrocellulose lacquers, varnishes and thinners, to dissolve the pigments for nail varnishes. It exhibits high dilution ratios with both aromatic and aliphatic diluents and is the least toxic of industrial organic solvents.

**Pharmaceuticals:** Ethyl acetate is an important component in extractants for the concentration and purification of antibiotics. It is also used as an intermediate in the manufacture of various drugs.

**Flavors and essences:** Ethyl acetate finds extensive use in the preparation of synthetic fruit essences, flavors and perfumes.

**Flexible packaging:** Substantial quantities of ethyl acetate are used in the manufacture of flexible packaging and in the manufacture of polyester films and BOPP films. It is also used in the treatment of aluminium foils.



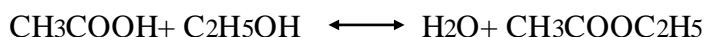
**Occurrence in wines:** Ethyl acetate is the most common ester found in wine, being the production of the most common volatile organic acid-acetic acid and the ethanol alcohol created during the fermentation of wine. The aroma of ethyl acetate is most vivid in younger wines and contributes towards the general perception of "fruitiness" in the wine. Sensitivity varies with most people having a perception threshold around 120 mg/lit. An excessive amount of ethyl acetate is considered a wine fault. Exposure to oxygen can exacerbate the fault due to the oxidation of ethanol creating acetaldehyde. This can leave the wine with sharp vinegar like taste.

**Miscellaneous:** Ethyl acetate is used in the manufacture of adhesives, cleaning fluids, inks, nail-polish removers and silk, coated papers, explosives, artificial leather, photographic films & plates.

In the field of entomology, ethyl acetate is an effective poison for use in insect collecting and study. In a killing jar charged with ethyl acetate, the vapors will kill the collected (usually adult) insect quickly without destroying it. Because it is not hygroscopic, ethyl acetate also keeps the insect soft enough to allow proper mounting suitable for a collection.

## 1.2 Production of Ethyl acetate:

Ethyl acetate is produced by the esterification reaction of ethyl alcohol and acetic acid using catalysts such as sulphuric acid, Para toluene sulphonic acid or ion exchange resins. The reaction of ethanol (EtOH) with acetic acid (AcOH) towards ethyl acetate (EtAc) and water (H<sub>2</sub>O) is an equilibrium reaction.



The achievable conversion in this reversible reaction is limited by the equilibrium conversion. Furthermore, the quaternary system of the above components is highly non ideal, and the formation of binary and ternary azeotropes compounds the complexity of the separation in conventional distillation columns. These facts suggest the suitability of reactive distillation for the production of ethyl acetate and have motivated several studies on the use of reactive distillation for this process.

### **1.3 Background of Reactive distillation process:**

Even after so many years distillation still remains one of the most important separation processes in chemical industry. All around the globe, in almost all the chemical industries a significant fraction of capital investment and operating cost involves distillation. As a result any kind of improvement of distillation operation can be very beneficial economically. Reactive distillation is one major step in the history of distillation in achieving these goals. The reactive distillation combines both chemical reaction and multi component separation into a single unit. Reactive distillation (RD), the combination of chemical reaction and distillation in a single unit operation, has proven to be advantageous over conventional process systems consisting of separate reactor and distillation units.

These processes as a whole are not a new concept as the first patent dates back to the 1920s<sup>1</sup>. The initial publications dealt with homogeneous self- catalyzed reactions such as esterifications and hydrolysis. Heterogeneous catalysis in reactive distillation is a more recent development and was first described by Spes<sup>2</sup>. While the concept existed much earlier, the first real- world implementation of reactive distillation took place in 1980s. The relatively large amount of new interest in reactive distillation is due to the numerous advantages it has over ordinary distillation. Enhanced reaction rates, increased conversion, enhanced reaction selectivity, heat integration benefits and reduced operating costs are just to name a few. All these factors contribute to the growing commercial importance of reactive distillation. However, since heat transfer, mass transfer, and reactions are all occurring simultaneously, the dynamics which can be exhibited by catalytic distillation columns can be considerably more complex than found in regular columns. This result in an increase in the complexity of process operations and the control structure installed to regulate the process.

### **Advantages of Reactive Distillation process:**

These advantages include:

- Capital cost savings from the elimination of unit operations.
- Improved selectivity and conversion.
- Potential to overcome chemical equilibrium limitations.
- Ability to react away azeotropes.
- And use of heat of reaction to reduce energy consumption through energy integration.

### **Disadvantages of Reactive Distillation process:**

However, there are drawbacks to reactive distillation which include:

- Incompatibility of process conditions for reaction and separation.
- Residence time requirements.
- Volatility constraints for the reagents and products in the reaction zone of the distillation column and
- Most importantly, the lack of well established design methods.

There are many documented success stories involving the industrial implementation of reactive distillation. The applications of reactive distillation in the chemical and petroleum industries have increased rapidly in the past decade (Taylor and Krishna, 2000). One such example is the manufacturing of methyl acetate by the Eastman Chemical Company. In this case a single reactive distillation column replaced the traditional flow sheet consisting of eleven major unit operations along with an assortment of heat exchangers, pumps and controllers. The result was a five-fold reduction in capital investment and energy consumption over the conventional design for methyl acetate production. In the present project, the study the dynamics and control of a reactive distillation column for the production of ethyl acetate will be discussed, which is one of the most widely, used esters.

### **1.4 Background of ASPEN PLUS software:**

In 1970s the researchers at MIT's Energy Laboratory developed a prototype for process simulation. They called it Advanced System for Process Engineering (ASPEN). This software has been commercialized in 1980's by the foundation of a company named AspenTech. ASPEN PLUS offers a complete integrated solution to chemical process industries. This sophisticated software package can be used in almost every aspect of process engineering from design stage to cost and profitability analysis. It has a built-in model library for distillation columns, separators, heat exchangers, reactors, etc. Custom or propriety models can extend its model library. These user models are created with FORTRAN subroutines or Excel worksheets and added to its model library. Using Visual Basic to add input forms for the user models makes them indistinguishable from the built-in ones. It has a built-in property databank for thermodynamic and physical parameters. During the calculation of the flow sheet any missing parameter can be estimated automatically by various group contribution methods.

Aspen Plus can interactively change specifications such as, flow sheet configuration, operating conditions, and feed compositions, to run new cases and analyze process alternatives Aspen Plus allows us to perform a wide range of tasks such as estimating and regressing physical properties, generating custom graphical and tabular output results, fitting plant data to simulation models, optimizing process, and interfacing results to spreadsheets.

### 1.5 ASPEN DYNAMICS:

In Aspen Dynamics we can easily:

- Add, change and remove control elements
- Select measured and manipulated variables from selection lists
- Configure cascade control loops
- Import control structures from other Aspen Dynamics generated input files

When an Aspen Dynamics simulation is created, level, pressure and temperature controllers are automatically included where appropriate. For flow-driven dynamic simulations, these controllers are configured as shown in this table:

For this type is used	This type of controller of control	To directly manipulate
Liquid level	Proportional only	Liquid flow rate
Pressure	Proportional integral	Vapor flow rate or duty, as appropriate
Temperature	Proportional integral	Duty

# CHAPTER-02

# LITERATURE REVIEW

The study of RD dates back as far as the early 1920s (Backhaus, 1921), but the idea did not draw significant interest until the advent of effective algorithms for the simulation and design of reactive distillation systems. There is a large number of examples of RD simulation found in the literature (Chang & Seader, 1988; Dudukovic and Lee (1998), Malone, & Doherty, 2000; Taylor, & Krishna, 2000 Nishith Vora and Prodromos Daoutidis 2001; Michael C. Georgiadis, Myrian Schenk, Efstratios N. Pistikopoulos, Rafiqul Gani 2002 ;). A number of papers and patents have explored the RD systems. The literature up to 1992 was reviewed by Doherty and Buzad (1992). Most of the papers were discussed by steady-state design and optimization problems. Only a few papers studied the dynamic of reactive distillation or the interaction between design and control.

Al-Arfaj and Luyben (2002), Sneesby et al. (1997), Kumar and Daoutidis (1999), discussed the decentralized PI control structures for reactive distillation column. Sneeby et al. (1998), Al-Arfaj and Luyben (2002) discussed the possibility of multiple steady states in many reactive distillation systems. The presence of multiplicities and the highly nonlinear nature of reactive distillation may impose limitations on use of linear controllers. Kumar and Daoutidis (1999) have discussed the superior performance of nonlinear controller compared to linear controller for reactive distillation systems.

Suzuki et al. (1971) described the design and simulation of an ethyl acetate reactive distillation column. A reactive column with thirteen equilibrium stages, made up of eleven trays, one total condenser and one reboiler, was simulated in the study. Ethyl acetate was withdrawn from the top in the distillate stream, and the feed consisting of the reactants acetic acid and ethyl alcohol was introduced on stage six. This single feed configuration for ethyl acetate reactive distillation has become a prototype configuration and many researchers have developed/applied several numerical algorithms to solve the nonlinear steady-state model for the above prototype configuration. The objective of above research was to establish the convergence characteristics and the robustness of the numerical algorithms, rather than to analyze the column behavior in detail. The ethyl acetate conversion (-30%) and the ethyl acetate purity of (=50%) reported in the literature (Suzuki et. al., 1971) for the single feed configuration. This ethyl acetate composition is lower than the azeotropic composition (54%). In practice, it would be desirable for the column at least attain product purity greater than the azeotropic composition to justify the use of a reactive distillation instead of a conventional configuration of a reactor followed by a distillation column.

The composition as well as reaction rate profiles for the single feed composition shows that majority of the reaction takes place in the reboiler and the bottom part of the column, and the upper half of the column acts essentially as a distillation column instead of reactive distillation column. This happens because the acetic acid, which is the heaviest of the four components, moves down the column and is present in negligible amount in the upper half of the column,

resulting in negligible reaction in the upper half of the column. The absence of the reaction in the upper part of the column hinders achieving a composition higher than the azeotropic composition with this configuration. Furthermore, it can be observed that ethanol is present in significant amounts in the upper half of the column, which retards the separation (and hence achievable purity) due to closeness of boiling points between the ethyl acetate and ethyl alcohol. These observations suggest that the countercurrent flow of the reactants ethyl alcohol and acetic acid in the column could enhance the forward reaction on several trays, thus improving the overall conversion. [Alejski et al. \(1996\)](#) studied the multiple feed configurations for an experimental setup for ethyl acetate column. The experimental results show an improvement in the conversion of ethyl acetate.

[Vora and daoutidis \(2001\)](#) studied the dynamics and control of ethyl acetate reactive distillation column. Initially, they proposed a configuration that involves feeding the two reactants in different trays and allows for the attainment of higher conversion and purity at the steady state than does the conventional configuration, which involves feeding in a single tray. Then, an analysis of the column dynamics is performed, which reveals the existence of fast and slow dynamics corresponding to the liquid flow rates and the liquid compositions and holdups. Further analysis of the tray hydraulics of the column shows that this time-scale multiplicity was induced by the relatively instantaneous approach of the volume of liquid on a tray to the weir volume. A model of the slow dynamics was obtained under the quasi-steady-state assumption of equilibrium between the liquid volume on a tray and the weir volume. The study of the column dynamics was followed by a detailed analysis of different control configurations. Specifically, control configurations with constant energy (steam) requirements and constant bottom flow rates are studied in terms of the effect of the reflux flow rate (a key manipulated input) on the product purity (a key controlled output), and the existence of input multiplicity in the column is demonstrated. Finally, model-based linear and nonlinear state feedback controllers, along with conventional SISO proportional integral (PI) controllers are designed, and their performance and robustness is evaluated through simulations.

[Georgiadis et al. 2002](#) used a high fidelity (hybrid) dynamic model that has been validated against experimental data and then studied the interactions of design and control in a reactive column involving the production of ethyl acetate from the esterification of acetic acid with ethanol ([Schenk et al., 1999](#)). The problem posed was a dynamic optimization problem and it is solved using control vector parameterization techniques. Two state-of-the-art optimization strategies are employed. In the first strategy, the design and control tasks are optimized *sequentially* while in the second; design and control are optimized *simultaneously*. The potential synergistic benefits of this simultaneous approach are investigated. In both cases, multi-loop proportional integral (PI) controllers are used. An economically attractive and fully operable reactive distillation and control scheme was obtained which exploited the interactions between process designs, process control and process operability.

# CHAPTER-03

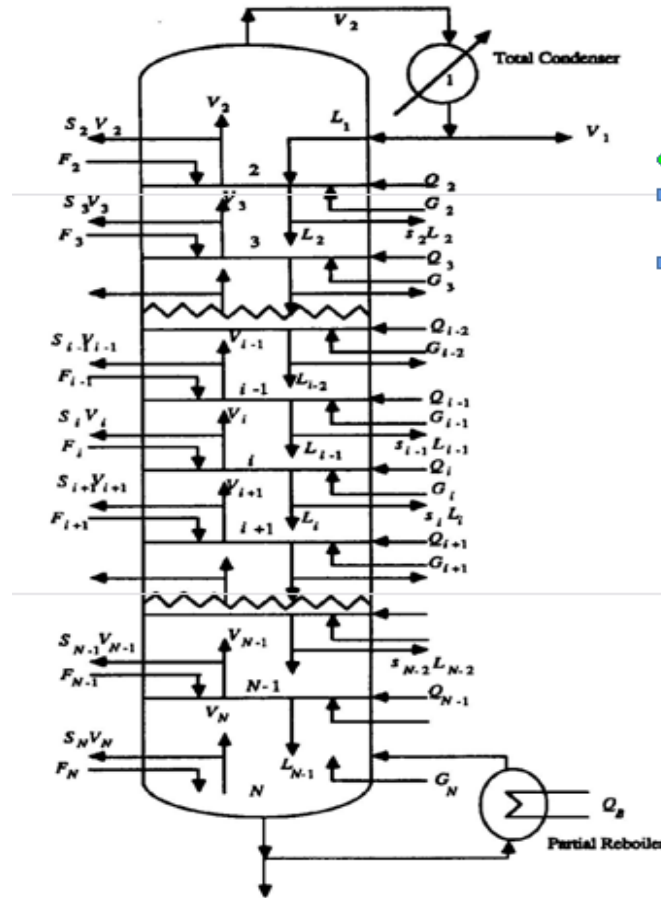
## MODELING



Consider an N stage column separating C stage components where stage 1 is total condenser and stage N is partial reboiler. All the chemical reactions occur in liquid phase.

Assumptions:

- The process has reached steady state.
- The liquid composition at each stage is homogeneous and equal to the composition of the liquid leaving the stage.
- The vapor and liquid leaving at any stage are in physical equilibrium.



Configuration of reactive distillation column

The three equations which describes the physical and chemical processes on stage 'i' are-

3.1 Component material balances:

$$M_{ij} = (1 + S_i) v_{ij} + (1 + S_i) l_{ij} - v_{i+1,j} - l_{i-1,j} - f_{ij} - g_{ij} - U_i \sum v_{jn} r_{in} \quad , \quad j = 1, \dots, C \quad (1)$$

$U_i \sum v_{jn} r_{in}$  accounts for chemical reactions.

### 3.2 Energy balances:

$$E_i = (1 + S_i) H_i + (1 + S_i) h_i - H_{i+1} - h_{i+1} - h_{Fi} - H_{Gi} - Q_i \quad (2)$$

Here unlike the component material balances, it is not necessary to take the heat of reaction into account because enthalpies are referred to the elements rather than to the components.

### 3.3 Equilibrium relationships:

$$Q_i = K_{ij} l_{ij} \frac{v_i}{L_i} - v_{ij}, \text{ where } c = 1, \dots, C \quad (3)$$

$Q_i$  is derived from the definition of  $K_{ij}$ .

$$K_{ij} = \frac{y_{ij}}{x_{ij}} \text{ or } K_{ij} x_{ij} - y_{ij} = 0 \quad (4)$$

$x$  and  $y$  represent the component mol fractions.

Specifications for equations (1-3) include the total number of stages, stage locations of all feeds, side streams, and heat exchangers, all stage pressures and liquid-phase holdup volumes, and complete specification of each fresh feed. This leaves  $N$  specifications that must be made before solving the  $N(2C + 1)$  equations for the corresponding  $N(2C + 1)$  iteration variables. If the  $N$  specifications are  $N$  stage heat transfer rates, then the equations are solved for  $2CN$  component molar flows and  $N$  stage temperatures. However, because condenser and reboiler duties are strongly dependent and can generally be specified independently over only narrow unknown ranges, other specifications are more desirable. The total condenser stage is a special case which is not represented by equation (3). Let the component molar flows of the liquid distillate be called  $v_{1,1}, v_{1,2}, v_{1,3}, \dots, v_{1,c}$ . If the reflux ratio is given, the top stage energy balance equation is changed to reflux ratio equation.

$$E_1 = L_1 - RV_1 \quad (5)$$

Where  $R$  is the reflux ratio.

The rate equations in terms of concentrations are

$$\begin{aligned} r_{in} &= \frac{dC_{in}}{dt} \\ &= \sum K_p \prod C_{qi}^{n_{qi}} \\ &= \sum A_p \exp(-E_p/RT_i) \prod C_{qi}^{n_{qi}} \end{aligned}$$

where  $C_{iq} = l_{iq} / (L_i v_i^L)$  is conc of component  $q$

The variable  $A$  is the Arrhenius pre exponential factor,  $E$  is the energy of activation,  $R$  is the universal gas constant and  $T_i$  is the temperature at stage  $i$ . This reaction model can deal with either irreversible or reversible reactions.

# CHAPTER-04

## STEADY STATE SIMULATION

#### 4.1 Single feed:

Single feed consists of a mixture of both acetic acid and ethanol and it is fed to the distillation column at a particular tray. In this present work we introduced the feed at stage 6.

##### Input data:

Number of stages: 13

Feed stage: 6

Feed temperature: 220 K

Pressure: 1 atm

Feed composition (mole flow):

CH<sub>3</sub>COOH - 53.8 lb mol/hr

ETOH - 53.8 lb mol/hr

Reflux rate: 10

Bottoms rate: 86.8 lb mol/hr

Reactive trays in column are from tray 2 to tray 12

Hold ups:

Stage No.	Liquid hold ups
1 - 12	80.6 cu ft
13	266.95 cu ft

First a radfrac column is selected from the model library in the aspen plus user interface. The above mentioned input data is given in the data browser. Then steady state simulation is executed and the results are obtained in the data browser itself. The typical flow sheet for the single feed is given in fig 1. The liquid composition profile is also given in fig 2.

#### 4.2 Double feed:

In double feed, unlike single feed we use two feed inputs. At first acetic acid is fed at stage 6 and ethanol at stage 7 and then we kept one feed stage constant let's say acetic acid feed stage is kept constant and change another and vice versa. The combination for which we get maximum purity of ethyl acetate composition is used for dynamics and control studies. In this work the best purity is obtained when acetic acid is fed at stage 2 and ethanol is fed at stage 12. These results are shown in Fig 3 and the corresponding flow sheet is also given in fig 4 and the liquid composition profile is given in fig 5.

#### **4.3 Effect of reflux ratio on the ETAC composition:**

In steady state simulation of ethyl acetate reactive distillation column, initially we kept the reflux ratio at 0.001 then we went on increasing it to 10. The liquid composition of ethyl acetate is also noted from every simulation and a graph (fig 6) is drawn between the reflux ratio and mol fraction of ethyl acetate. It is observed that at reflux ratio of 4 we are getting maximum purity for ethyl acetate compared to other reflux ratios.

#### **4.4 Effect of bottoms rate on the ethyl acetate composition:**

In steady state simulation of ethyl acetate reactive distillation column, initially we kept bottoms rate at 1 lb mol/hr and then we went on increasing it to 105 lb mol/hr. the liquid composition of ethyl acetate is also noted and a graph (Fig7) is drawn between the bottoms rate and mol fraction of ethyl acetate. It is observed that at 100 lb mol/hr we are getting maximum purity for ethyl acetate in the distillate.

# CHAPTER-05

## DYNAMICS

The obtained steady state flow sheet and results are exported to aspen dynamics. When exported a pressure controller is introduced by the aspen dynamics itself. This pressure controller controls the pressure in the reboiler. In addition to that we have placed 2 level controllers (one for condenser level and one for sump level) and 2 flow controllers for the two feed inputs. The flow controllers are in reverse action as if any disturbance in flow is controlled automatically by the changing its valve opening. Later a composition controller is placed for controlling ethyl acetate composition at the distillate and also at the bottoms. The flow sheet of the process in which composition control of ethyl acetate in the distillate is given in Fig 8 and the flow sheet for composition control of ethyl acetate in the bottom is given in fig 9.

For the control of ethyl acetate mol fraction in the distillate using a PID controller, we used condenser duty as manipulated variable. The steady state ethyl acetate mol fraction in the distillate was 0.5613. We have given a set point of 0.6 and a gain of 0.5. The set point is obtained at 1.7hrs. As we go on increasing the gain we are attaining the set point in less time. For all the cases we used servo problem i.e. changing the set point to 0.6 from steady state value of 0.5613. For a set point of 0.62 the liquid is being vented out from the condenser. The graph from fig 10 to fig 13 shows the response of composition controller for ethyl acetate in the distillate at various gain values.

For the control of ethyl acetate mol fraction in the bottoms using a PID controller, we used reboiler duty as manipulated variable. The steady state ethyl acetate mol fraction in the bottoms was 0.1163. We have given a set point of 0.05 and for a gain of 0.5 and 1 response of the controller were shown in fig 14 and fig 15. We can clearly see the oscillations in the graph. Now a closed loop feedback relay test is done from which we get the optimum gain and integral time. The method used for tuning is tyreus-luyben. The response of the controller after the tuning of the controller has been done is given in fig 16. For the bottoms controller a dead time of 1 min is given to account for the dynamic lags that are always present in any real controller.

# CHAPTER-06

## RESULTS



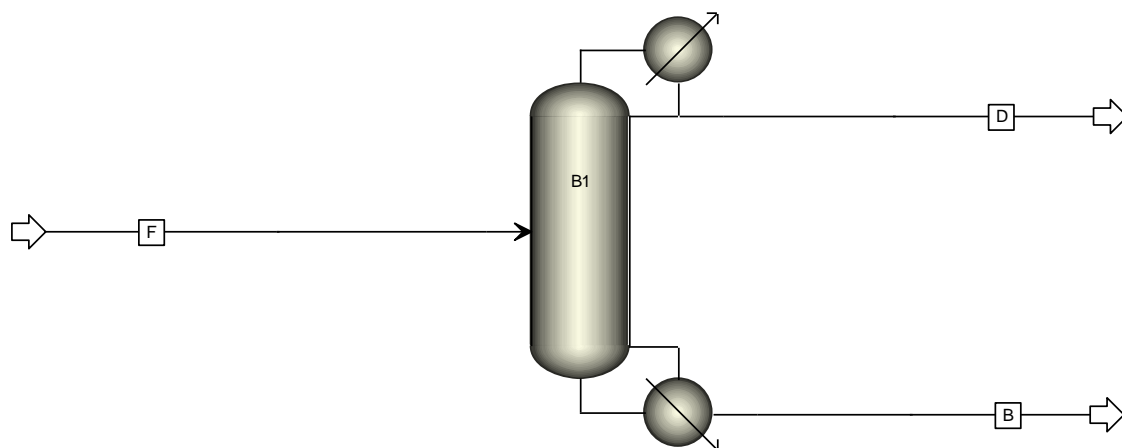


Fig 1 - Single feed flow sheet in ASPEN

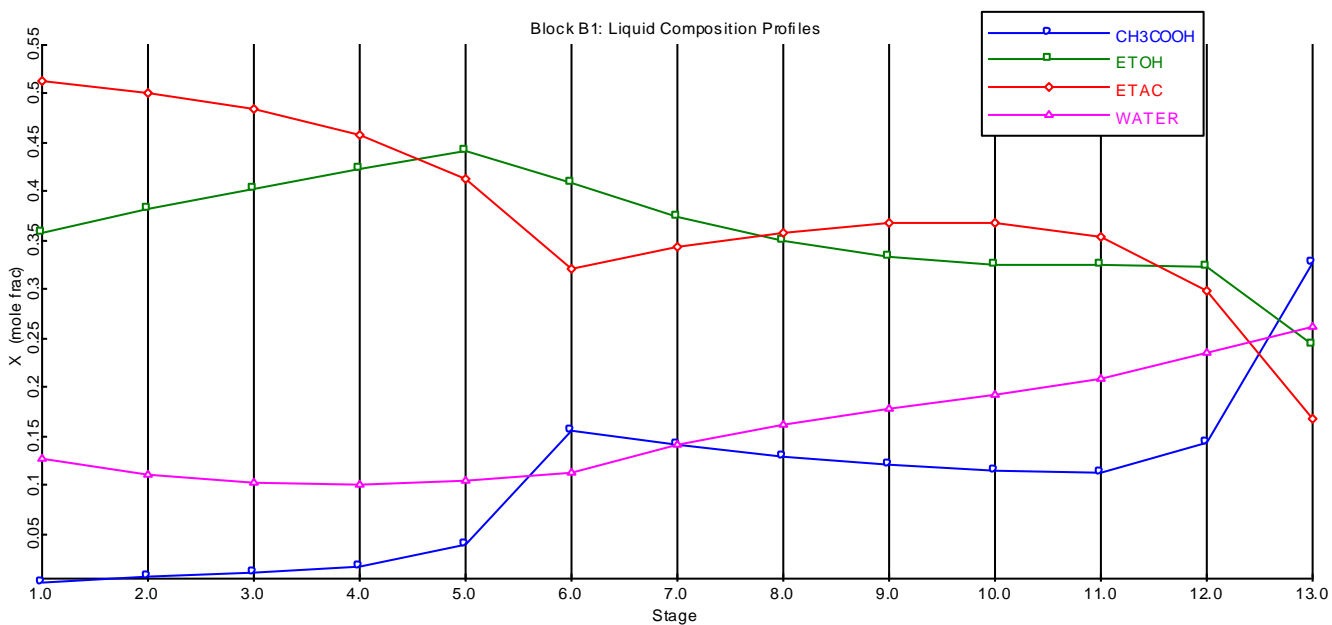


Fig 2 - Single feed composition profile

<i><b>FEED 1 (CH<sub>3</sub>COOH)</b></i>	<i><b>FEED 2 (ETOH)</b></i>	<i><b>ETAC MOLE FRACTION</b></i>
TRAY 2	TRAY 4	0.512
TRAY 2	TRAY 5	0.532
TRAY 2	TRAY 6	0.5466
TRAY 2	TRAY 7	0.556
TRAY 2	TRAY 8	0.5627
TRAY 2	TRAY 9	0.5673
TRAY 2	TRAY 10	0.5707
TRAY 2	TRAY 11	0.5733
<b>TRAY 2</b>	<b>TRAY 12</b>	<b>0.5753</b>
TRAY 3	TRAY 12	0.5623
TRAY 4	TRAY 12	0.5387
TRAY 5	TRAY 12	0.5243
TRAY 6	TRAY 12	0.5167

Fig 3 - Optimum tray for second feed

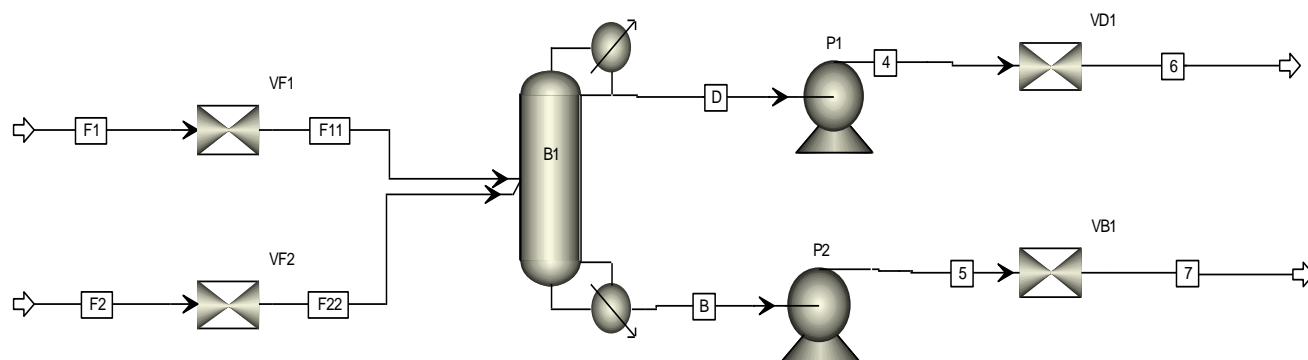


Fig 4 - Double feed flow sheet in ASPEN

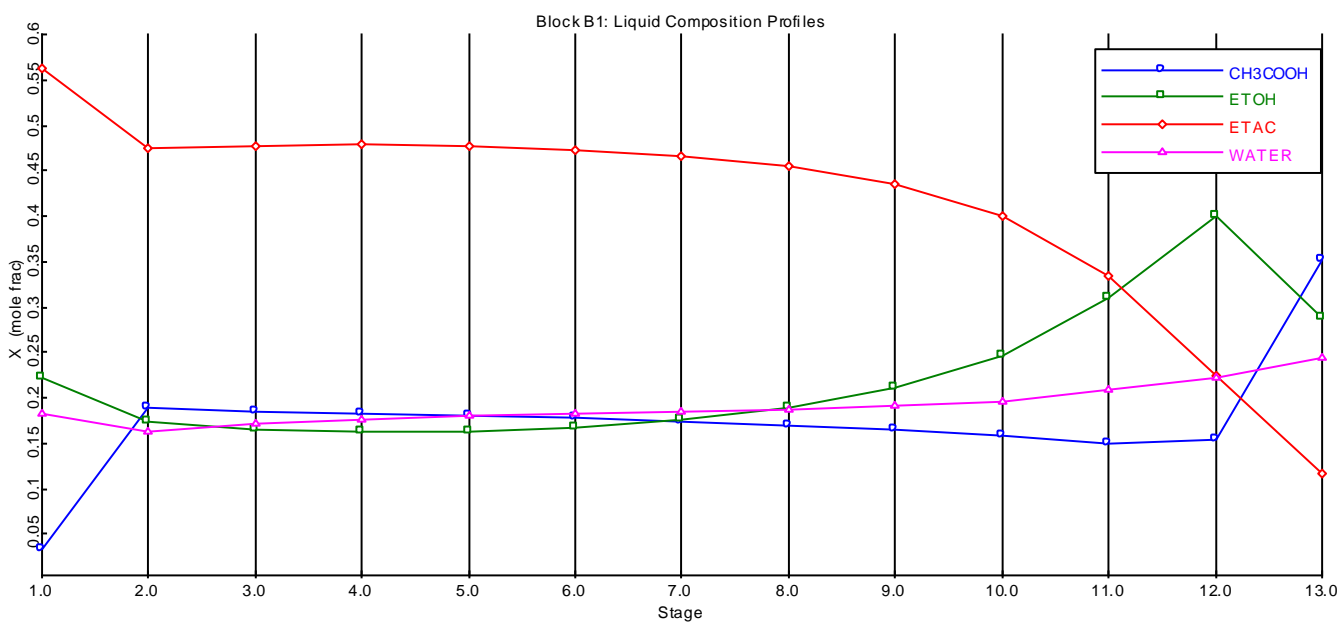


Fig 5 - Double feed composition profile

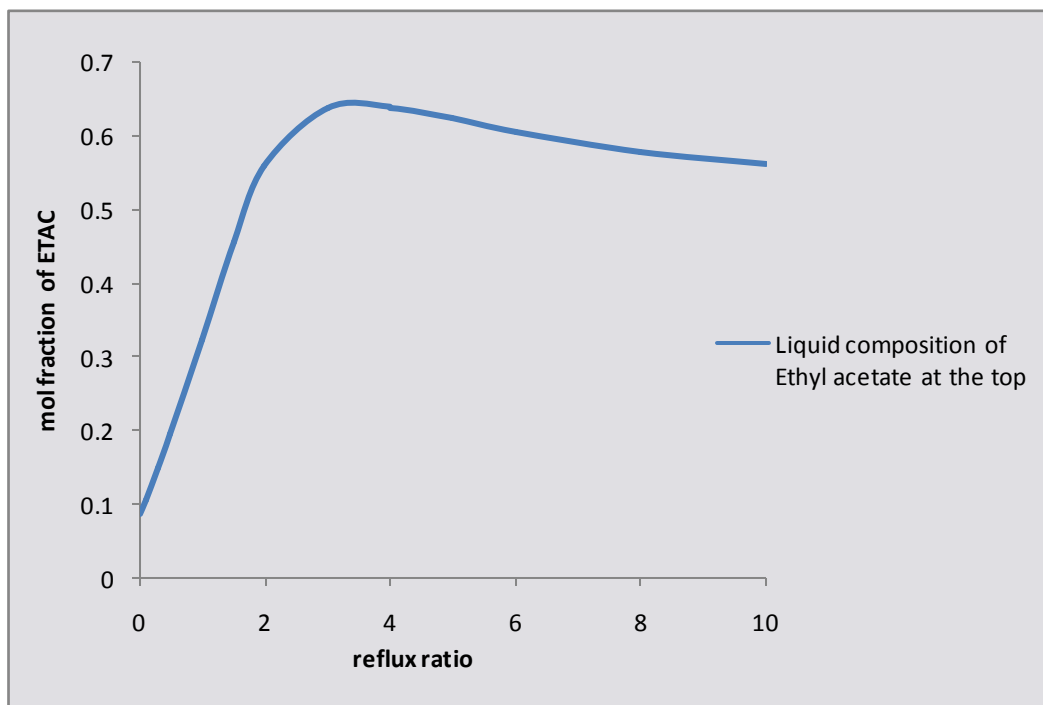


Fig 6 - Effect of reflux ratio on the ETAC composition

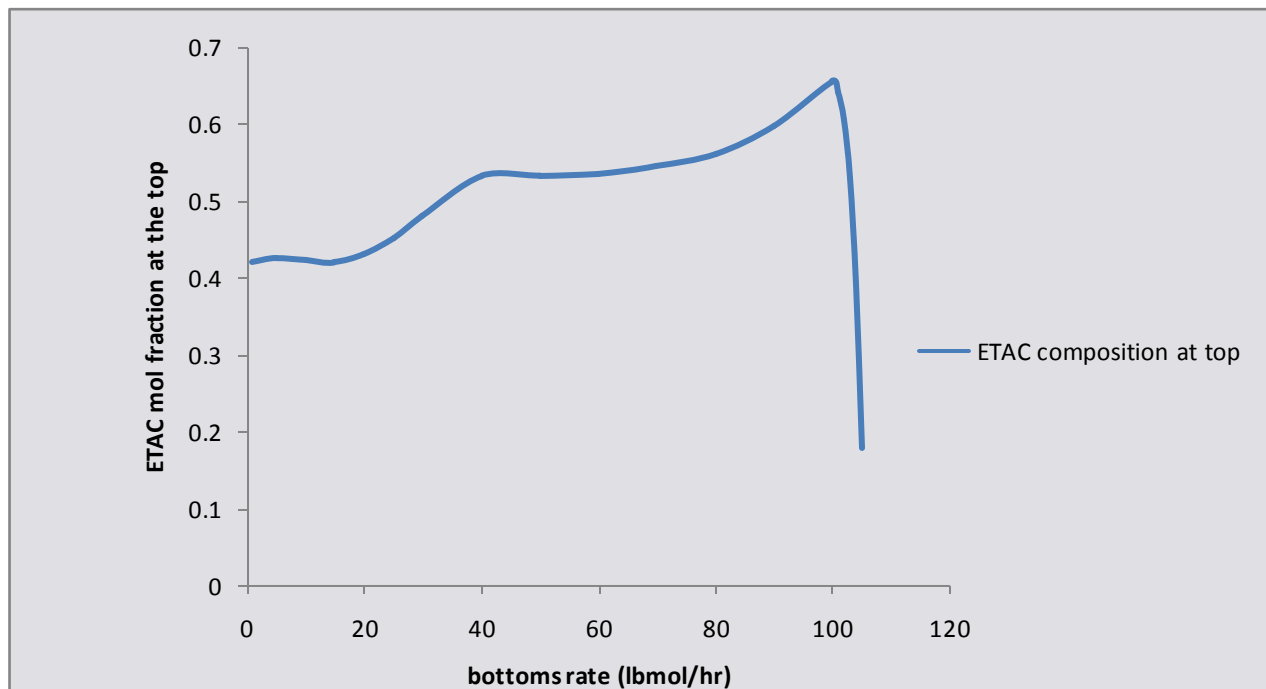


Fig 7 – Effect of bottoms rate on the ETAC composition

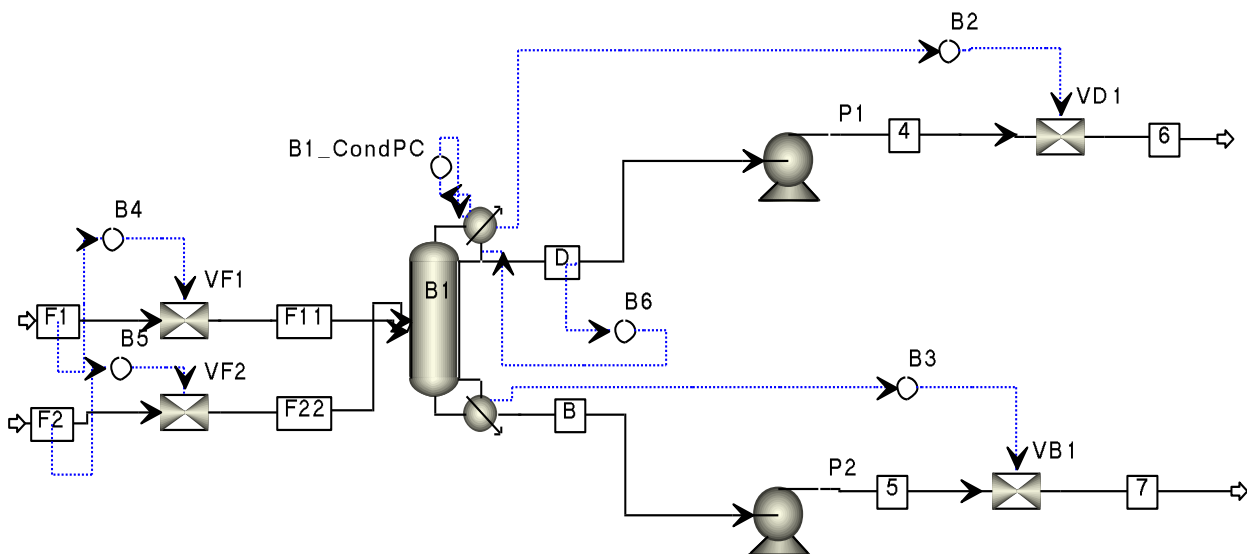


Fig 8 - Composition control for ethyl acetate in distillate

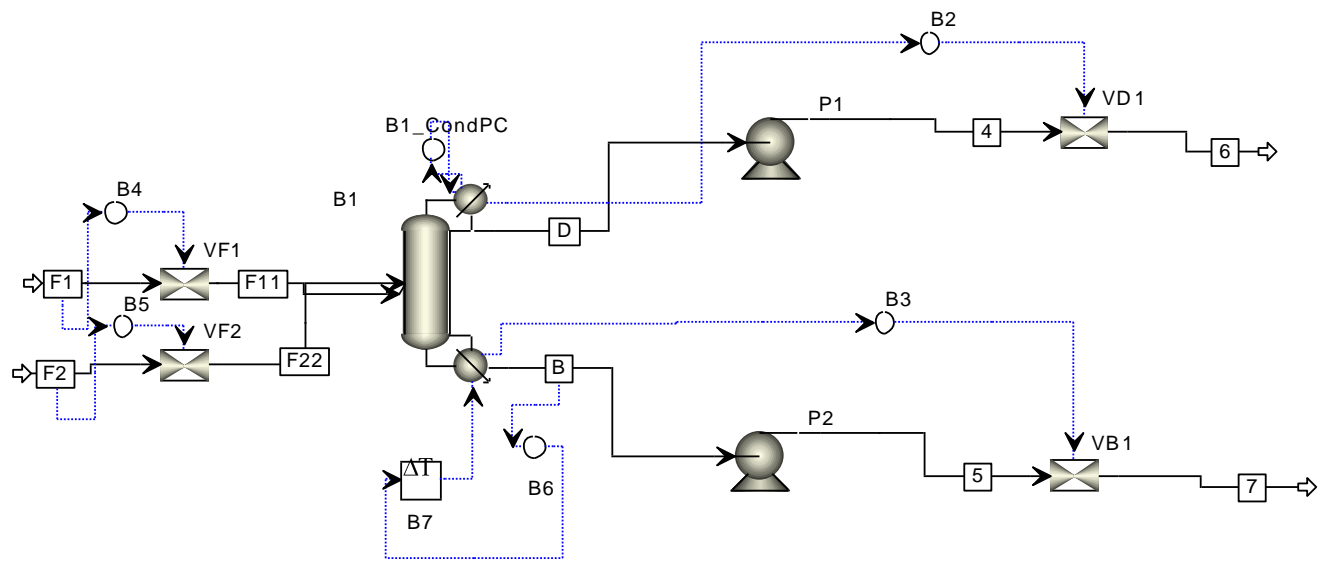


Fig 9 - Composition control for ethyl acetate in bottoms

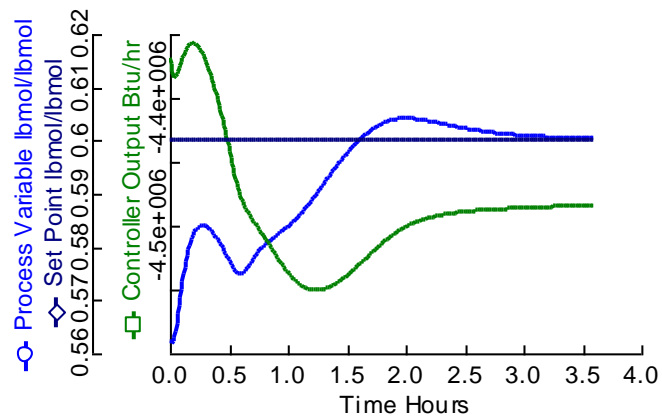


Fig 10 - Response of controller when  $K_c = 0.5$

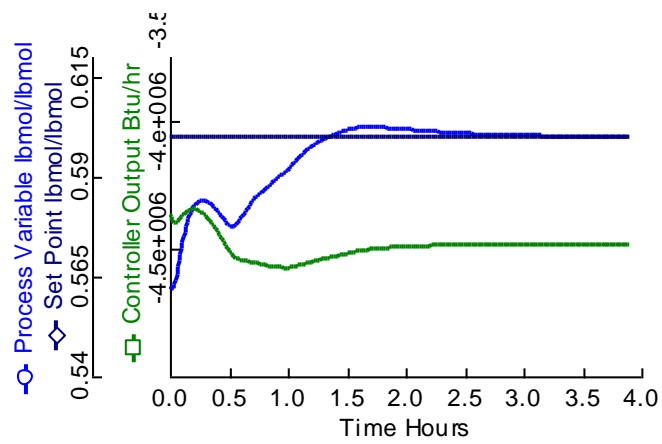


Fig 11 - Response of controller when  $K_c = 1$

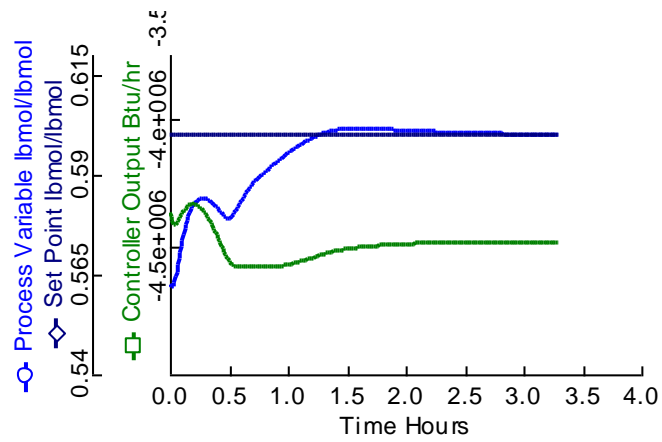


Fig 12 - Response of controller when  $K_c = 1.5$

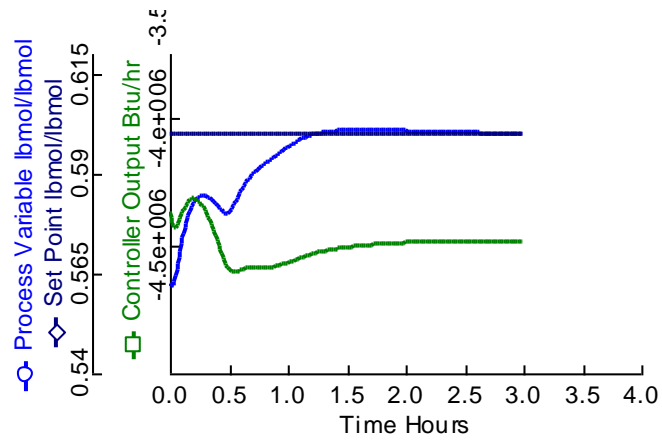


Fig 13 - Response of controller when  $K_c=2$

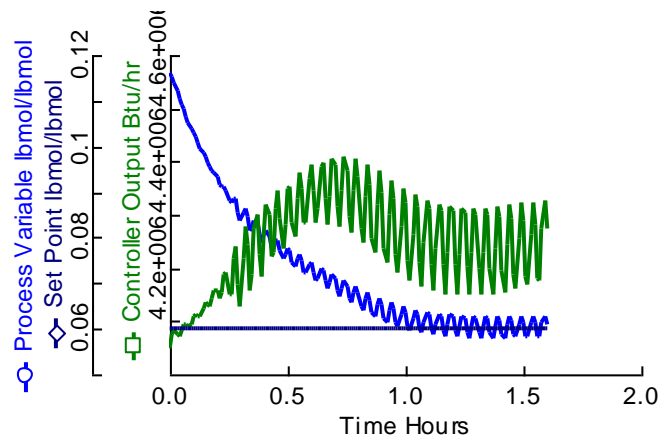


Fig 14 - Response of controller for XB when  $K_c = 1$

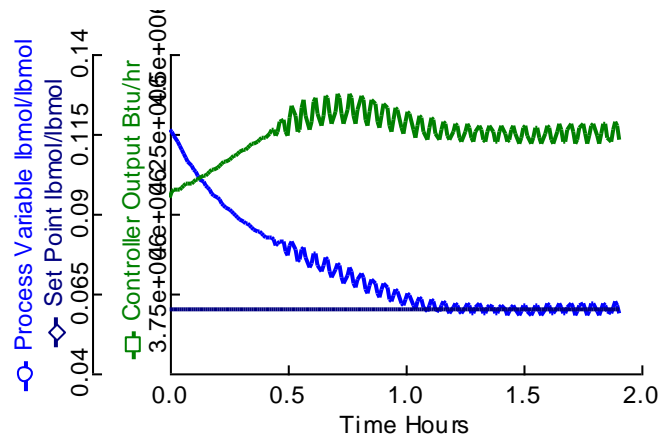


Fig 15 - Reponse of controller for XB when  $K_c=0.5$

### After relay feedback test (closed loop test)

Method used: tyreus-luyben  
 Controller type- PI  
 Gain = 0.160007  
 Integral time = 7.92 min  
 Set point 0.05 from 0.116

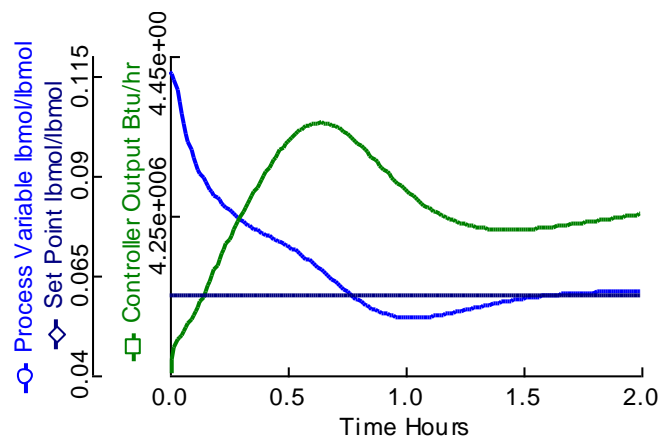


Fig 16 – Response after tuning the controller



## CONCLUSION:

It is clearly known that double feed gives more purity of ethyl acetate than single feed. The optimum feed location was located where acetic acid is fed at tray 2 and ethanol at tray 12. Effect of reflux ratio and bottoms rate on the ethyl acetate composition in the distillate is shown. Control of XD is done by using condenser duty as manipulated variable, where liquid is being vented for a set point of 0.61 or greater than that for the ethyl acetate distillate composition. Control of XB is done by using reboiler duty as manipulated variable, the tuning of controller is done by using tyreus-luyben closed loop method and optimum gain and integral time was found.

# CHAPTER-07

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